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(54) Title: RADIATION CURABLE INTUMESCENT COATINGS

(57) Abstract: An intumescent coating composition comprises a resin system together with at least one photo-initiator and/or sensitiser. The photo-initiator and/or sensitiser serves to initiate curing of the intumescent coating composition when exposed to actinic radiation.



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### **Radiation Curable Intumescent Coatings**

The present invention relates to radiation curable intumescent coatings.

Intumescent coating compositions are commonly used to protect structural steel components in buildings. Such intumescent coatings contain various ingredients  
5 which, when exposed to heat, react together to produce an insulating foam known as 'intumescent char'. This char greatly reduces the rate of heating experienced by the steel, thus prolonging the integrity of the steel and extending the time taken for a building to collapse, thereby allowing additional time for safe evacuation of a building.

10 Generally, steel is known to lose half of its load bearing capacity at around 550°C. Tests conducted under cellulosic fire conditions i.e. the heating regime found in non-oil burning fire situations, have shown that the failure point for steel is around 550°C. Therefore, this is often referred to as the 'critical failure temperature'.

During a fire situation a steel structure will gradually heat up, the rate of  
15 heating being dependent upon the specific dimensions of the steel substrates used in the structure. The rate of heating of a given steel substrate is dependent upon the  $H_p/A$  value of the section where  $H_p$  is the perimeter of the steel substrate when viewed in cross-section, and  $A$  is the cross sectional area of the steel substrate. The configuration of steel components can also influence the rate of heating.

20 Therefore, a steel substrate with a large perimeter ( $H_p$ ) will receive more heat than one with a small perimeter. However, the greater the cross sectional area ( $A$ ), the more heat the steel substrate can absorb. Thus, a large relatively thin steel substrate having a high  $H_p/A$  value will heat up more quickly than a thicker steel substrate having a lower  $H_p/A$  value.

The thickness of coating that is applied may depend upon the  $H_p/A$  value of the steel, its configuration, and the level of fire protection required (typically 30 minutes to 2 hours). These variables mean that the thickness of coating applied can vary from 250 $\mu$ m to 5mm. Typically, an intumescent coating designed for cellulosic fire conditions is applied at a coating thickness that, in a fire situation, will swell to between 25 to 50 times the original coating thickness.

Intumescent coatings designed for cellulosic fire protection tend to be based on thermoplastic acrylic and vinylic resins which also include a relatively high proportion of organic solvent to facilitate application to a steel substrate. However, such coatings are inherently slow drying. This is a particular problem when coating thicknesses of up to 2mm may be applied to the steel substrate in a single coat. Stringent legislation governing organic solvent emissions has lead to greater use of water-based resins but slow drying remains a problem.

Traditionally intumescent coatings were applied to steel substrates on-site during the construction phase of a building. However, in recent years there has been a trend for a number of major users of intumescent coatings to move to the off-site or 'in-shop' application of coatings to steel substrates. This allows greater control of the application conditions. In order to facilitate the maximum throughput of steel substrates through application shops, it is of the utmost importance that the coating is dry enough to be handled in the shortest possible length of time. In response to this need, coating compositions have been developed using more volatile solvents. However, these can still take up to 24 hours to become sufficiently dry to be handled without damaging the coating. The use of ovens can reduce drying times, though

these are expensive to operate, and due to the thermoplastic nature of the coatings it is necessary for cooling to occur prior to handling in order to prevent damage.

Intumescent coatings exist that are based on thermosetting resin systems. These tend to be two-pack epoxy systems which contain little if any solvent relative to thermoplastic intumescent coatings and as such have considerably shorter drying  
5 times. However, it is necessary to apply a much thicker coating to the steel substrate in order to achieve a comparable level of fire protection as would be obtained with a thermoplastic coating and therefore, may not be deemed economical.

That said, it would be of enormous benefit to 'in-shop' applicators for drying  
10 to be as quick as possible, ideally as part of a continuous process of application, drying and removal of substrates prior to shipping to site.

The present invention has been made from a consideration of this problem.

According to the present invention there is provided an intumescent coating composition, wherein the coating composition comprises a resin system together with  
15 at least one photo-initiator and/or sensitiser, said photo-initiator and/or sensitiser serving to initiate curing of the intumescent coating composition when exposed to actinic radiation

Advantageously, steel substrates that are coated with the coating composition of the present invention 'dry to handle' much more rapidly than prior art intumescent  
20 materials. Drying times of around 24 hours, or even longer, are reduced to around 2 minutes, although this varies slightly in accordance with the thickness of the coating. Therefore, significant time spent waiting for coated steel substrates to dry is eliminated, as is the use of costly ovens and therefore the cooling time associated therewith.

The resin system preferably constitutes from 20% to 70% by weight of the coating composition, and most preferably from 35% to 45% by weight of the coating composition.

The resin system of the present invention generally comprises one or more  
5 oligomers and/or monomers, the former being relatively high in molecular weight and viscosity, and the latter being relatively low in molecular weight and viscosity. These materials are selected in such a manner as to take into account various parameters such as adhesion of the composition to a substrate (both as a dry film and under fire conditions), application viscosity, flexibility, hardness and water sensitivity. These  
10 materials in the context of the present invention will cure by way of exposure to actinic radiation in the presence photo-initiators and/or photosensitisers.

Although these molecular species are able to polymerise in the presence of actinic radiation and photo-initiator(s) two different reaction mechanisms are possible depending upon the resin system used, namely free radical polymerisation or cationic  
15 polymerisation. The resin system may be cured solely by one of these reaction mechanisms or may consist of a combination of the two.

For free radical cure the oligomers that may be employed usually have a degree of unsaturation present, preferably by way of ethylenically unsaturated bonds and more preferably these unsaturated bonds will be present as  $\alpha$ - $\beta$  ethylenically  
20 unsaturated carboxylate ester groups. Suitable species include but are not limited to any of the following groups:- acrylates, methacrylates, unsaturated polyesters, unsaturated polyamides, vinyls (such as vinyl esters), olefins, unsaturated ketones, azides and halogen modified saturated polymers or any combination thereof. The functionality of these species may vary from 1 to 6, preferably from 1 to 2, and is

most preferably 1 i.e. mono-functional. Mono-functional oligomers generally give rise to a cured polymer coating that is thermoplastic in nature. Such coatings tend to have the required flow properties during a fire situation, thus enabling a good depth of char to be produced.

5            Preferably the oligomer(s) of the resin system are acrylic and suitable species include any of, but are not limited to, epoxy acrylates (aromatic or aliphatic), aromatic epoxy acrylates, acrylated oil epoxy acrylates, novalac epoxy acrylates, aliphatic epoxy acrylates, urethane acrylates (aromatic and aliphatic), polyester acrylates, amine modified polyether acrylates, silane acrylates, silicone acrylates and acrylic  
10 acrylates or any combination thereof. Most preferably the acrylate oligomers are aromatic epoxy acrylates and most preferably monofunctional aromatic epoxy acrylates.

The acrylate oligomers preferably have molecular weights from 300 to about 10000.

15            For free radical cure monomers that may be employed generally have a degree of unsaturation and include, but are not limited to species which include any of the following groups:- acrylic, methacrylic, vinylic or allylic.

Suitable vinylic species include, but are not limited to any of styrene, vinyl ethers, vinyl acetate, vinyl pyrrolidone and vinyl caprolactam and combinations  
20 thereof.

Suitable methacrylic species include, but are not limited to any of alkyl methacrylates, glycol methacrylates, diol methacrylates and ethoxylated/propoxylated methacrylates any combination thereof.

Suitable allylic species include, but are not limited to allyl cyanurates and allyl ethers and combinations thereof.

Preferably the monomer(s) of the resin system are acrylic species and include, but are not limited to any of the following:- aromatic, aliphatic and cycloaliphatic acrylates, alkoxylated acrylates, acrylate, esters of alcohol or glycols, ether acrylates and acrylic acid. Preferably the acrylate monomer is a long chain aliphatic acrylate monomer or an alkoxylated acrylate monomer and most preferably a long chain aliphatic monofunctional acrylate such as isodecyl or lauryl acrylate or a monofunctional alkoxylated acrylate monomer. The acrylate monomers preferably have functionality of from 1 to 5, preferably from 1 to 2 and most preferably of 1 i.e. monofunctional. Mono-functional monomers assist in the maintenance of thermoplasticity within the cured coating. The acrylate monomers may have a molecular weight of from about 100 to about 1200.

It is necessary to consider the nature of the alkoxylation of the acrylate monomer as this affects the hydrophilicity of the intumescent coating composition. Therefore, the alkoxylated acrylate monomer is preferably ethoxylated or propoxylated. More specifically, the most preferred acrylate monomer is ethoxylated nonyl phenol acrylate.

Where used the monomer component(s) is used primarily to reduce the viscosity of the intumescent coating composition. However, excess monomer can result in lower durability and poor cure, particularly at the surface of the intumescent coating composition.

The cationic cure oligomer(s) that may be included in the present invention preferably include, but are not limited to any of:- cycloaliphatic epoxies, aromatic

epoxies, epoxy novolacs, hydroxyl functional compounds such as alcohols, glycols, polyether/polyester polyols, epoxidised polybutadienes and vinyls or any combination thereof. The functionality of the species may be monofunctional or they may be multifunctional. For cationic cure the monomers used preferably include, but are not  
5 limited to any of: vinyl ethers, oxetanes, and cycloaliphatic epoxies or any combination thereof.

The oligomer component(s) preferably constitute from 75 to 100% by weight of the resin system.

The monomer component(s) preferably constitutes from 0% to 25% by weight  
10 of the resin component composition and more preferably from 5% to 15% by weight of the resin.

Preferably the resin system should be capable of being cured by radiation comprising ultra-violet and near-visible radiation having an output in the range of from 200nm to 450nm and more preferably from 350nm to 450nm. Such radiation  
15 may be obtained using a gallium doped metal halide lamp. Most preferably the resin system should be capable of being cured by ultra-violet radiation via a free radical mechanism.

As mentioned previously, photo-initiators and/or sensitisers are required in order to facilitate cure in the radiation sensitive oligomers and monomers. These  
20 should be selected on the basis of the mechanism of the curing process and on other parameters such as pigmentation and thickness of the coating to be cured.

For free radical curing the free radicals are generated by two mechanisms following two different pathways, i.e. unimolecular or bimolecular pathways.



Preferably the photo-initiators of the present invention react unimolecularly, the reaction involving an  $\alpha$ -cleavage step.

Suitable photo-initiators for free radical curing, which may be used alone or in combination, preferably include any of the following:-  $\alpha$ -hydroxy ketones, phosphine  
5 oxides,  $\alpha$ -amino ketones, benzoin and derivatives, benzil ketals, benzoin ethers, acetophenone and derivatives, phenylglyoxylates, formates, benzophenone and derivatives and thioxanthone and derivatives.

Preferably the photo-initiators for free radical curing of the present invention are phosphine oxides, examples of which include bis(2,4,6-trimethyl benzoyl)-phenyl  
10 phosphine oxide, (2,4,6-trimethyl benzoyl)-diphenyl phosphine oxide and (2,4,6-trimethyl benzoyl)-ethoxyphenyl phosphine oxide and  $\alpha$ -hydroxy ketones, examples of which include 2-hydroxy-2-methyl-1-phenyl-(propan-1-one and 1-hydroxy-cyclohexyl-phenyl-ketone.

The most preferred photo-initiators of the present invention are bis(2,4,6-  
15 trimethylbenzoyl)-phenyl phosphine oxide and 2-hydroxy-2-methyl-1-phenyl-propan-1-one.

Advantageously, the liquid 2-hydroxy-2-methyl-1-phenyl-propan-1-one can be used as a solvent for any of the solid phosphine oxide photo-initiators mentioned above thereby facilitating easy incorporation of such photo-initiators.

20 For cationic curing the photo-initiators that may be used in the present invention preferably include, but are not limited to any of ferrocenium salts, onium salts, triarylsulphonium salts, hexafluoroantimonate salts, iodonium salts and diazonium salts or combinations of any the said materials.

An onium salt photo-initiator may optionally be used as a cationic photo-initiator even where the predominant curing mechanism is via a free radical route due to the fact that di-vinyl ether is capable of being cured by both the free radical and cationic mechanisms.

5           The total level of the photo-initiator(s) used preferably constitutes from 0.1% to 8% by weight of the resin component and more preferably from 0.5% to 5% by weight of the resin component. If employed, the cationic photo-initiator should preferably be used with the free radical photo-initiator(s) at a ratio of between 1.3:3.1 by weight.

10           That said, the level of inclusion of the photo-initiator(s) may depend on the photo-initiator(s) employed for example where the composition of the present invention comprises bis(2,4,6-trimethyl benzoyl)-phenyl phosphine oxide, the said photo-initiator preferably constitutes from 0.1% to 5% by weight of the resin component and more preferably from 0.25% to 2% by weight of the resin component.

15           Where the composition of the present invention comprises a mixture of photo-initiators, namely bis(2,4,6-trimethyl benzoyl)-phenyl phosphine oxide and 2-hydroxy-2-methyl-1-phenyl-propan-2-one the said mixture preferably constitutes from 0.1% to 10% by weight of the resin component and more preferably from 1% to 4% by weight of the resin component.

20           The intumescent coating compositions of the present invention also contain intumescent ingredients that can react together in the presence of heat from a fire to form a protective insulating intumescent char. As well as being correctly blended to produce the required degree/quality of intumescent char the intumescent ingredients

should also ideally be of low solubility to ensure that the coating is as durable as possible.

The solubility of the intumescent ingredients should be less than 10g per 100ml of water, and preferably less than 1g per 100ml of water.

5       The intumescent ingredients preferably constitute from 30% to 80% by weight of the coating composition and more preferably from 45% to 70% by weight of the coating composition.

10       The intumescent ingredients preferably comprise at least one acid source, at least one carbon source, and at least one gas source (spumific) together with a nucleating agent.

Under the influence of heat (typically 200°C – 300°C) the acid source decomposes to produce acid which then proceeds to react with the carbon source to yield large amounts of carbon. Simultaneously, under the influence of heat (typically 200°C – 400°C) the gas source decomposes to produce gas which results in the  
15   bubbling or swelling of the coating. The combined action of the acid and the gas results in the formation of an intumescent char or foam.

Suitable acid sources include, for example, melamine phosphate, magnesium sulphate and any boric acid or any combination thereof. In addition ammonium polyphosphate may also be used which can be coated or uncoated. Coating  
20   ammonium polyphosphate reduces its solubility in water, resulting in improved durability. Furthermore, coating ammonium polyphosphate can result in lower viscosity of the coating due to a reduction in the resin absorbing properties of the ammonium polyphosphate. The coating may be, for example, epoxy, melamine formaldehyde or silane.

The most preferred acid source is ammonium polyphosphate, and is ideally coated with melamine formaldehyde or silane.

The acid source preferably constitutes from 30% to 80% by weight of the intumescent ingredients and more preferably from 35% to 65% by weight of the  
5 intumescent ingredients.

Suitable carbon sources include, for example, polyhydric alcohols such as pentaerythritol, di-pentaerythritol, tri-pentaerythritol, starch and expandable graphite.

The preferred carbon sources are pentaerythritol, di-pentaerythritol and combinations thereof. The carbon source preferably constitutes from 5% to 30% by  
10 weight of the intumescent ingredients are more preferably from 5% to 25% by weight of the intumescent ingredients.

Suitable gas sources include, for example, melamine, melamine phosphate, melamine borate, melamine cyanurate, tris-(hydroxyethyl) isocyanurate (THEIC), ammonium polyphosphate and chlorinated paraffin.

15 The preferred gas source is melamine. The gas source preferably constitutes from 5% to 35% by weight of the intumescent ingredients.

Although not essential for the intumescent reaction to take place, nucleating agents are an important preferred ingredient since they promote sites for the intumescent char to form. These materials are generally inert, inorganic materials and  
20 may include, for example, silica, silicates, mica and bentonite clay. The preferred nucleating agent is silica.

Titanium dioxide has been traditionally used as a nucleating agent in intumescent coating compositions, particularly since it also provides colour to the coating. However, titanium dioxide has a high refractive index (RI) and as such

absorbs radiation throughout the entire ultra-violet spectrum. Therefore, the use of titanium dioxide in the present invention would prevent the ultra-violet component of the actinic radiation reaching the resin component giving rise to poor curing of the coating. Silica has a low RI and as such is relatively transparent to UV radiation.

5 Thus, preferably the nucleating agent of the present invention has an RI below 1.6 and more preferably an RI of below 1.5.

The nucleating agent preferably constitutes from 1% to 25% by weight of the intumescent ingredients and more preferably from 5% to 15% by weight of the intumescent ingredients.

10 The intumescent ingredients of the present invention preferably have a particle size below 100 microns in order to obtain satisfactory dispersion in the coating.

Optionally, wetting agents (dispersion aids) may be added to the coating composition to facilitate dispersion. These agents may be either reactive or non-reactive.

15 For example, reactive wetting agents for free radical systems contain ethylenically unsaturated bonds and thus can co-react with the ethylenically unsaturated bonds of the resin component. Non-reactive wetting agents do not contain reactive groups that can react with any other ingredients, and particularly those of the resin component.

20 Suitable wetting agents include, for example, proprietary acrylate wetting agents, polysiloxanes, ethoxylates and soya lecithin.

The preferred wetting agent is soya lecithin. The wetting agent constitutes from 0% to 2% by weight of the coating composition.

Further additional additives may be optionally included in the intumescent coating composition of the present invention which include, for example, at least one flame retardant to aid char formation, at least one metal salt to improve char characteristics, and at least one fibre filler to strengthen the char and prevent char  
5 degradation.

Suitable flame retardants include, for example, compounds containing either phosphorus, chlorine, bromine or nitrogen or combinations thereof. Specific examples include ammonium polyphosphate, triphenyl phosphate, tricresyl phosphate, chlorinated paraffin, tris-(2-chloroethyl) phosphate and tris-(2-hydroxyethyl)  
10 isocyanurate. Further possible flame retardants include inorganic compounds such as aluminium and magnesium hydroxides.

The flame retardant preferably constitutes from 0% to 80% by weight of the intumescent ingredients.

Suitable metal salts which include, for example zinc borate, zinc stannate,  
15 aluminium hydroxide, magnesium hydroxide and antimony oxide and mixtures thereof.

The preferred metal salts are zinc borate and aluminium hydroxide. The metal salt(s) preferably constitute from 0% to 10% by weight of the intumescent ingredients.

Suitable fibre fillers include, for example, silica fibre, ceramic fibre and  
20 proprietary intumescent fibre. The fibre filler(s) preferably constitute from 0% to 10% by weight of the intumescent ingredients.

Once applied to a steel substrate the coating composition may be cured by the use of an arrangement of flood-lamps, moveable lamps or by passing the coated steel

substrate through a tunnel lined with appropriate lamps. Lamps having an output from 100W to 2000W are preferable.

Thus and in accordance with the second aspect of the present invention there is provided a method comprising the use of actinic radiation to cure an intumescent coating composition comprising a resin system together with at least one photo-initiator and/or sensitiser, said photo-initiator and/or sensitiser serving to initiate curing of the intumescent coating composition.

The coated steel substrate may be exposed to the radiation for between 30 seconds and 5 minutes, and at a distance from the radiation source of between 10cm and 2m. Depending upon the thickness of the intumescent coating composition, the total energy required for curing will range from  $0.5\text{Jcm}^{-2}$  to  $6.0\text{Jcm}^{-2}$  for UVV light ( $\lambda=395\text{nm}-450\text{nm}$ ) and from  $1.5\text{Jcm}^{-2}$  to  $9.0\text{Jcm}^{-2}$  for UVA light ( $\lambda=320\text{nm} - 395\text{nm}$ ). Filters may be employed to limit the output of the lamp to 350nm and above.

The amount of radiation required to cure the intumescent coating compositions increases with increasing thickness of coating applied, thus it should be appreciated that a very large number of combinations of lamp output, dwell times and distances from the light source are possible.

According to the third aspect of the present invention there is provided a method for the production of the intumescent coating composition described herein. This method preferably includes the steps:- mixing the intumescent ingredient(s) with the resin system and adding the photo-initiator(s) and/or sensitisers to the mixture,

Care must be taken to exclude sources of reactive radiation such as sunlight.

The coating composition of the present invention is applied to the steel substrate by methods well known to those skilled in the art, for example, airless spray, brush, roller, trowel and dipping. Preferably application will occur by airless spray. Airless spray methods require the use of airless spray pumps having a ratio of 45:1 or  
5 greater, and preferably 60:1 and a minimum air pressure of greater than 60psi and preferably 80psi. The compositions are preferably sprayed using a tip size ranging from 0.021 inch to 0.035 inch.

Where the viscosity of the composition of the present invention is high (greater than 0.8PaS @ 25°C – measured using a Bohlin CVO rheometer at a shear  
10 rate of 100s<sup>-1</sup>) due to high levels of intumescent ingredients, the coating, airless spray pump, and other equipment may be pre-heated prior to application/use to a temperature of from 40°C to 90°C. The exact temperature is dependent on the viscosity of the coating composition.

The coating composition of the present invention may be applied in liquid  
15 form to steel substrates up to several metres in length with a gauge thickness typically ranging from 5mm to 30mm whereby the thickness provides adequate fire protection for at least 30 minutes.

Steel substrates requiring fire protection are normally blast cleaned prior to the application of an intumescent coating to remove millscale and other deposits that may  
20 lead to premature failure of the intumescent coating either on prolonged atmospheric exposure or during a fire situation. In order to prevent deterioration of the blast cleaned surface, particularly where there is a delay in applying the intumescent coating, it is normal to apply a primer coating. This is often the case when the intumescent coating is applied on site.



Suitable primers include, for example, those based on epoxy, modified epoxy (such as modified with PVB) polyurethane, acrylic, vinyl and chlorinated rubber. Primers based on epoxy are preferred.

5 Additionally, particularly for in-shop application the primer may be based on radiation cured technology, to minimise the drying time of the primer and enable a rapid process. These coatings may be based on either free radical or cationic curing technology or a combination of the two. Primers based on free radical curing resin systems such as epoxy acrylate, urethane acrylate and polyester acrylate are preferred. The thickness of the primer can vary from 15 microns to 250 microns. Preferably the  
10 thickness should be in the range from 25 microns to 100 microns.

A topcoat may also be applied to the cured intumescent coating compositions of the present invention particularly to provide colour to exposed steelwork. A topcoat if correctly formulated will also enhance the durability of the intumescent coating compositions. A clear sealer may also be suitable.

15 Suitable topcoats include, for example, those based on epoxy, polyurethane, alkyd, acrylic, vinyl and chlorinated rubber. Topcoats based on polyurethane are preferred. Additionally, as with the primer, the topcoat may be based on radiation cured technology. Radiation cured topcoats based on urethane acrylate are preferred.

In order that the present invention may be more readily understood specific  
20 examples thereof are set out below:-

**Example Formulation A:**

<b>Component</b>	<b>Parts by weight:</b>
Ammonium polyphosphate (1)	32.50
Pentaerythritol (2)	8.50
Melamine (3)	11.50
Silica Flour (4)	6.25
Monofunctional aromatic acrylate oligomer (5)	36.00
Monofunctional aliphatic acrylate monomer –Isodecyl acrylate (6)	4.00
Free radical photoinitiator (7)	0.25
Wetting/Dispersing agent	1.00
<b>TOTAL</b>	<b>100.00</b>

**Example Formulation B:**

<b>Component</b>	<b>Parts by weight:</b>
Ammonium polyphosphate (1)	32.50
Pentaerythritol (2)	8.50
Melamine (3)	11.50
Silica Flour (4)	6.25
Monofunctional aromatic acrylate oligomer (5)	36.00
Monofunctional ethoxylated acrylate monomer – ethoxylated (4)	
Nonylphenol acrylate (8)	4.00
Free radical photoinitiator (7)	0.25
Wetting/Dispersing agent	1.00
<b>TOTAL</b>	<b>100.00</b>

- (1) FRCROS 489 (MF treated) from Great Lakes Chemical Company
- (2) Pentaerythritol Mono from Perstop Speciality Chemicals
- (3) Melamine grade 003 from BASF UK Ltd.
- (4) Silica flour HPF 3 from Sibelco Minerals and Chemicals
- 5 (5) CN131 from Cray Valley
- (6) Photomer 4810F from Cognis
- (7) Irgacure 819 from Ciba Speciality Chemicals
- (8) Photomer 4003 from Cogni

10 **Example Formulation C**

A commercially available thermoplastic acrylic and solvent based intumescent material, which has been included for comparison purposes.

Formulations A and B are manufactured by high speed dispersion (e.g. Greaves mixer) by first adding oligomer, monomer, photo-initiator and wetting agent  
15 and stirring well. The intumescent pigments are then added to this blend, stirring well until fully dispersed.

Formulations A, B and C were applied by drawdown onto 0.8mm thick, 150mm x 100mm sheet steel panels which had been sandblasted to ensure adequate adhesion of the coating. A thickness of 1.2mm was produced in each case. A gap of  
20 approximately 15mm wide was left uncoated along the 100mm side of the panel.

Formulations A and B were subjected to UV light from a gallium doped bulb using a filter which screens out wavelengths below 350nm. The panel was placed 20cm from the light source and exposed to the light for a period of 2 mins. This gives

a total energy output of  $1.8\text{Jcm}^{-2}$  for UVV light and  $2.6\text{Jcm}^{-2}$  for UVA light. These coatings were left for 1 hour prior to testing for their insulative properties.

Formulation C was applied and then left to dry for 3 days at  $35^{\circ}\text{C}$  so that the solvent could be eliminated from the coating.

5        The formulations were tested by means of application of a Meker burner onto the centre of the coated side of each panel, the burner being at a distance of 5cm from the panel. The temperature at centre of the uncoated back of each panel was measured in order to assess the insulative properties of each coating. An uncoated panel was included as a control in order to demonstrate the rate at which bare steel will heat  
10    during the same conditions.

The maximum depth of char as measured after 10 mins application of the burner were as follows:-

Formulation A – 22mm

Formulation B – 37mm

15        Formulation C – 35mm

The temperatures as measured on the back of the panels coated with each of the formulations A, B and C are set out in the graph, as shown in the accompanying fig. 1.

It is to be understood that the above described examples are given by way of example only. Many modifications and variations are possible.

## Claims

1. An intumescent coating composition, wherein the coating composition  
5 comprises a resin system together with at least one photo-initiator and/or sensitiser,  
said photo-initiator and/or sensitiser serving to initiate curing of the intumescent  
coating composition when exposed to actinic radiation.
2. A composition according to claim 1, wherein the resin system constitutes from  
20% to 70% by weight of the coating composition.
- 10 3. A composition according to claim 1 or claim 2, wherein the resin system  
comprises from 35% to 45% by weight of the coating composition.
4. A composition according to any preceding claim, wherein curing occurs via a  
free radical mechanism and/or a cationic mechanism.
5. A composition according to any preceding claim, wherein curing occurs via a  
15 free radical mechanism.
6. A composition according to any preceding claim, wherein the resin system  
comprises one or more oligomers and/or monomers.
7. A composition according to claim 6, wherein the oligomer has functionality in  
the range 1 to 6.
- 20 8. A composition according to claim 6 or claim 7, wherein the oligomer is  
selected from any of the following either alone or in combination:- acrylates,  
methacrylates, unsaturated polyesters, unsaturated polyamides, vinyls, olefins,  
unsaturated ketones, azides and halogen modified saturated polymers.

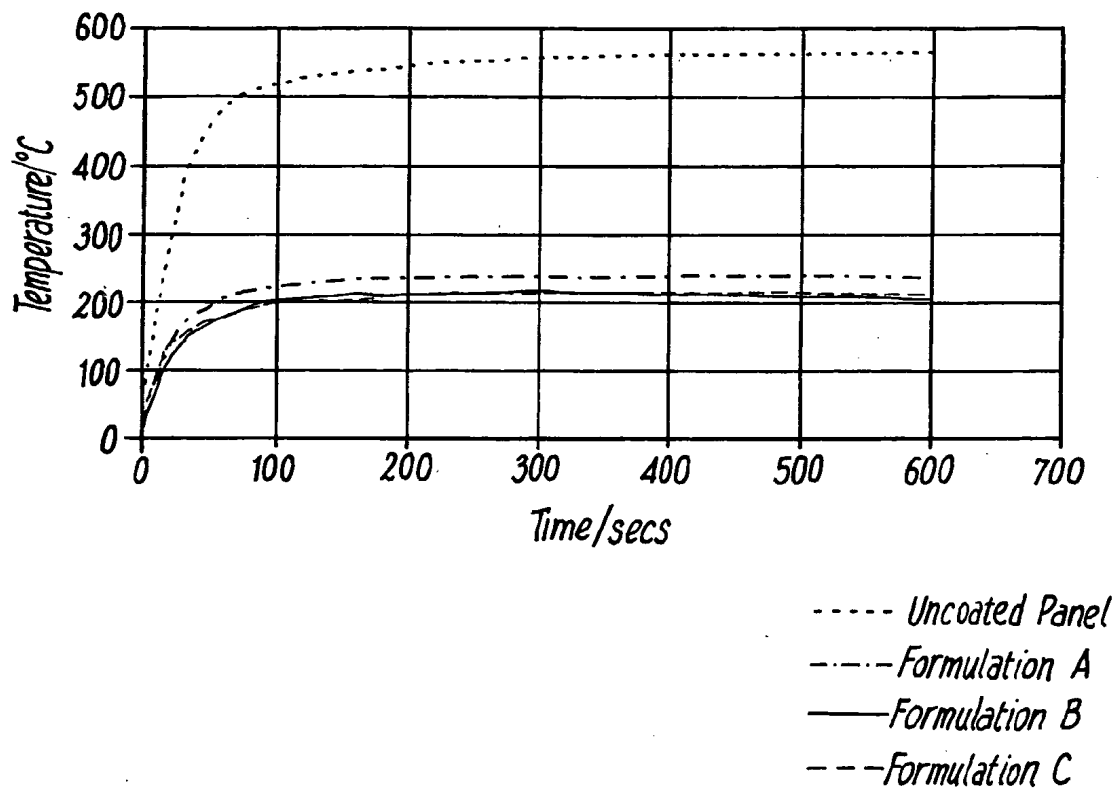
9. A composition according to any of claims 6 to 8, wherein the oligomer is acrylic.
10. A composition according to claim 9, wherein the said acrylic oligomer includes any of the following either alone or in combination:- modified and  
5 unmodified aliphatic and aromatic epoxy acrylates, phenolic (Novalac) epoxy acrylate, modified or unmodified aliphatic and aromatic urethane acrylates, modified or unmodified polyester acrylates, modified or unmodified polyether acrylates, acrylic acrylates, silicone acrylates and silane acrylates.
11. A composition according to claim 9 or claim 10, wherein the acrylic oligomer  
10 is a mono-functional aromatic epoxy acrylate.
12. A composition according to any of claims 9 to 11, wherein the oligomer constitutes from 75% to 100% by weight of the resin system.
13. A composition according to claim 6, wherein the monomer is selected from any of the following either alone or in combination:- acrylic, vinylic, methacrylic and  
15 allylic species.
14. A composition according to claim 13, wherein the monomer is acrylic.
15. A composition according to claim 13, wherein the acrylic monomer includes any of the following either alone or in combination:- aromatic, aliphatic and cycloaliphatic acrylates, alkoxyated acrylates, acrylate, esters of alcohol or glycols,  
20 ether acrylates and acrylic acid.
16. A composition according to claim 13 or claim 14, wherein the acrylic monomer is ethoxylated nonyl phenol acrylate.
17. A composition according to claim 14, wherein the monomer constitutes from 0% to 25% by weight of the resin system.

18. A composition according to any of claims 5, 13 to 17, wherein the monomer constitutes from 5% to 15% by weight of the resin system.
19. A composition according to any preceding claim, wherein the photo-initiator reacts unimolecularly.
- 5 20. A composition according to any preceding claim, wherein the photo-initiator includes any of the following either alone or combination:-  $\alpha$ -hydroxy ketones, phosphine oxides,  $\alpha$ -amino ketones, benzoin and derivatives,  $\alpha$ -formyl ketals, acetophenone and derivatives, phenylglyoxylates,  $\alpha$ -formyls, benzophenone and derivatives and thioxanthone and derivatives and mixtures thereof.
- 10 21. A composition according to any preceding claim, wherein the photo-initiator is a phosphine oxide.
22. A composition according to any preceding claim, wherein the photo-initiator is a mixture comprising a phosphine oxide and an  $\alpha$ -hydroxy ketone.
23. A composition according to any preceding claim, wherein the photo-initiator  
15 constitutes from 0.1% to 8% by weight of the resin system.
24. A composition according to any preceding claim, wherein the photo-initiator constitutes from 0.5% to 5% by weight of the resin system.
25. A composition according to any preceding claim, wherein curing occurs via ultra-violet radiation.
- 20 26. A composition according to any preceding claim, wherein the composition comprises intumescent ingredients which include any of the following:- at least one acid source, at least one carbon source, at least one gas source and mixtures thereof.
27. A composition according to claim 26, wherein the intumescent ingredients comprise from 30% to 80% by weight of the coating composition.

28. A composition according to claim 26 or claim 27, wherein the intumescent ingredients constitute from 45% to 70% by weight of the coating composition.
29. A composition according to claim 26, wherein the acid source includes any of the following:- melamine phosphate, magnesium sulphate, a boric acid, ammonium polyphosphate and mixtures thereof.
30. A composition according to claim 29, wherein the acid source is ammonium polyphosphate.
31. A composition according to any of claims 26 to 30, wherein the acid source constitutes from 30% to 80% by weight of the intumescent ingredients.
32. A composition according to claim 26, wherein the carbon source include any of the following:- pentaerythritol, di- pentaerythritol, tri-pentaerythritol, starch, expandable graphite and mixtures thereof.
33. A composition according to claim 26, wherein the carbon constitutes from 5% to 30% by weight of the intumescent ingredients.
34. A composition according to claim 26, wherein the gas source includes any of the following:- melamine, melamine phosphate, melamine borate, melamine cyanurate, tris-(hydroxyethyl) isocyanurate (THEIC), ammonium polyphosphate, chlorinated paraffin and mixtures thereof.
35. A composition according to claim 26, wherein the gas source constitutes from 5% to 35% by weight of the intumescent ingredients.
36. A composition according to any preceding claim, wherein the composition comprises at least one nucleating agent.
37. A composition according to claim 36, wherein the nucleating agent is silica.



38. A composition according to claim 36 or claim 37, wherein the nucleating agent constitutes from 1% to 25% by weight of the intumescent ingredients.
39. A composition according to any preceding claim, wherein the composition comprises at least one flame retardant.
- 5 40. A composition according to claim 39, wherein the flame retardant is a compound comprising phosphorus, chlorine, bromine, nitrogen and mixtures thereof.
41. A composition according to claim 39 or claim 40, wherein the flame retardant constitutes from 0% to 80% by weight of the intumescent ingredients.
42. The use of actinic radiation to cure an intumescent coating composition
- 10 comprising a resin system together with at least one photo-initiator and/or sensitiser, said photo-initiator and/or sensitiser serving to initiate curing of the intumescent coating composition.



**FIG. 1**

## INTERNATIONAL SEARCH REPORT

Intern: I Application No

PCT/GB 03/00520

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 C09D5/18

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

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## INTERNATIONAL SEARCH REPORT

International Application No

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